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**(54) A Method of Manufacture of
Perfumed Detergents**

(57) The aim of the invention is to achieve long-termed durability of scent of powdered detergents, especially during long storing, and to decrease the amount of perfume added to the detergent.

The aim is achieved by sorption of the perfume on a porous carrier which is mixed directly with the powdered

detergent and uniformly distributed therein. A sorbent with large active surface is used as a porous carrier, which sorbent is on the basis of polyacrylates, polyvinyl chloride and polyalkyl methacrylate and other similar compounds. The invention can be used in the production of pre-washing and soaking agents and detergents and in the production of perfumed cosmetic preparations and deodorants in powdered form.

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SPECIFICATION
A Method of Manufacture of Perfumed Detergents

The invention relates to a method of manufacture of perfumed detergents, especially powdered ones, washing and cleaning agents, stabilization of the perfume ingredient of these products during their storing being solved by decreasing its volatility and enhancing its resistance to chemical changes.

Known powdered washing and cleaning agents have been produced by mixing an atomized aqueous suspension of tensides, mainly of the anion-active type, and admixtures, in atomizing towers under heating or at cold, that is admixtures such as alkaline polyphosphates, alkaline silicates, sodium sulphate, sodium tetraborate, sodium carbonate, organic chelating agents on the basis of sodium salts of nitriletriacetic or ethylenediamine tetraacetic acid, carboxymethyl cellulose, optical refining compounds. Dry half-product is then homogenized in mixing devices with further powdered, or liquid ingredients, for example sodium peroxoborate, polyphosphates, alkaline proteases and amylases, non-ionogenic types of tensides, which ingredients are usually less stable in water and when dried at higher temperatures, optionally it is economically advantageous to add them directly into the product without preceding dissolving in water. A powdered detergent produced in this way is perfumed usually in the last production phase by atomizing liquid perfume composition by jet onto the product which is being mixed. Known processes for producing cleaning agents have been based on a similar principle, which processes further use during drying of the atomized solution or suspension higher content of inorganic ingredients, which are able to bind water from the solution or suspension, and dry product, usually in the form of a powder of different structure and shape of particles is perfumed by spraying with atomized or dispersed form of perfumed composition. The drawback of this process for producing detergent is that already in the course of production partial volatilization of odouriferous substances into atmosphere takes place and direct spraying of powdered detergent with perfumed composition does not bring about uniform product homogeneity as far as perfume content is concerned due to different sorption ability of individual detergent ingredients. Another drawback is that perfume losses are markedly increased during storing by its volatilization, or by the influences of chemical changes of its individual ingredients, which can alter the original scent in character and intensity. This results in a substantially lower efficiency of perfume exploitation in powdered detergents. A larger amount of perfume has to be added in order to keep the desired useful properties of detergents after a long storing time and this means uneconomical use of a higher perfume content in

production. Powdered detergents with enhanced perfume stability are known, in which the stability is obtained by the presence of amorphous silicic acid hydrate in a powdered detergent added to the perfume in a known manner. The residual perfume content after six months of storing is at these detergents between 30 and 50% by weight as compared with a detergent without ingredient of enhancing perfume stability, where the residual perfume content of approximately 10% by weight was found after the same period. The drawbacks of such a detergent and a process for its manufacture are analogous to the foregoing processes because the perfume is again sprayed onto a powdered detergent containing in addition amorphous silicic acid hydrate which does not ensure its defined, regulated and decreased desorption during storing.

The drawbacks mentioned are eliminated according to the invention by a process for producing detergents, especially powdered washing and cleaning agents, in which process individual ingredients of the agent are mixed in the desired ratio, perfume is added and the mixture is homogenized. According to the invention the perfume is sorbed before its addition to the other ingredients on porous carrier, advantageously on the basis of hydrated silicon dioxide, titanium dioxide, polyethylene acrylate, polyvinyl chloride, polymethyl acrylate, polymethyl methacrylate, acetylcellulose, polyphenylene oxide, polyethylene terephthalate, polyamide, urea-formaldehyde resin, melamine-formaldehyde resin, copolymers of acrylonitrile, butadiene and styrene, polyacrylonitrile, copolymer of vinyl chloride and vinyl acetate, polyethylene, polypropylene, polystyrene and their mixtures. The sorbed perfume content is added to the powdered detergent in the amount of 0.05 to 10% by weight, advantageously 0.2 to 1% by weight, and is perfectly homogenized. The perfume content is 10 to 65% by weight in this sorbent.

The method of manufacture of detergents according to the invention utilizes the excellent properties of perfumed sorbents residing in stability of the perfume composition sorbed on the porous carrier, which stability is determined by its defined, regulated and substantially decreased desorption. The process further uses physical and physicochemical properties of the composition which properties enable direct mixing with powdered detergent which leads to a product in which the perfume ingredient is uniformly distributed on the porous carrier in the whole volume of the powdered detergent after homogenization of the mixture. The advantage of the process for producing detergent according to the invention compared with known processes lies thus in that the perfumed sorbent having desired particle size can be modified by the content of the perfume composition so that its physical properties, especially its bulk weight, correspond to the bulk weight of the powdered detergent and enables its simple mixing with the

detergent at obtaining homogeneous product which is stable during long-termed storing. The properties of the sorbent further stabilize the perfume composition sorbed at it, which
5 manifests itself by a substantial reduction of its volatility and by the avoidance of chemical changes in its composition during the storing of the powdered detergent resulting in changes in the character of its scent. Another advantageous
10 property of porous sorbents containing a perfume is that they make the intensity and character of scent of the original perfume composition more expensive.

The effect of the method according to the
15 invention lies thus in that, by the addition of the perfume composition sorbed on a porous carrier before its mixing with the other ingredients, a high intensity of scent on the porous carrier is obtained even after long-termed storing which
20 leads to a substantial enhancement of perfume efficiency and to keeping the original useful properties. The effect of the process for producing detergents according to the invention lies further in that by decreasing perfumation of the
25 powdered detergent during production by 50 to 60% by weight, equal or even higher scent intensity of the product can be obtained after a longer storing time as compared with known processes of detergent perfumation.

30 The method of manufacture of powdered detergents according to the invention is further illustrated by several examples.

Example 1

A powdered detergent prepared by mixing
35 39.5% by weight of a first mixture produced by drying in an atomizing tower using heat, which mixture contained 25% by weight of surfactants on the basis of sodium dodecylbenzene sulphonate and 2% by weight of carboxymethyl
40 cellulose, 40% by weight of tripolyphosphate, 6% by weight of sodium silicate, 1.5% by weight of optically whitening agents, 25.5% by weight of sodium sulphate, and 60.2% by weight of a second mixture, containing 30% by weight of a
45 hard soap of higher fatty acids, 60% by weight of sodium carbonate and 10% by weight of sodium sulphate, with 0.3% by weight of a perfumed sorbent which contained 50% by weight of porous hydrated silicon dioxide and 50% by
50 weight of a perfume composition on the basis of eucalyptus oil, benzoate and toluate derivatives, terpinylacetate, dehydrolinacetate and aldehydes and ketones and their derivatives. Homogeneous detergent with intensive characteristic scent both
55 in solid state and after dissolving in water is obtained. The detergent prepared according to this example retained practically the original intensity and character of scent of its perfume composition after 12 months of storing.

60 Example 2

95% by weight of a powdered detergent, prepared in a manner similar to that described in Example 1, was mixed with 5% by weight of a

perfumed sorbent which contained 97% by
65 weight of porous hydrated silicon dioxide and 3% by weight of the perfume composition from Example 1. Properties of the thus prepared detergent were the same as in the Example 1 as regards stability of the perfume composition after
70 12 months storing.

Example 3

99.7% by weight of a powdered detergent, prepared in a manner similar to that described in Example 1, was mixed with 0.3% by weight of a
75 perfumed sorbent which contained 67% by weight of a porous sorbent on the basis of polyethylene terephthalate and 33% by weight of a perfume composition on the basis of limonene, dipentene and veratrene. A homogeneous
80 detergent of an intensive and characteristic scent was obtained. After 12 months storing the thus prepared detergent had the same properties as that made according to Example 1.

Example 4

85 A powdered washing detergent was prepared by the usual production process for preparation of powdered detergents, which detergent had the following composition: 8% by weight of sodium dodecylbenzene sulphonate, 5% by weight of a
90 hard soap of higher fatty acids, 27% by weight of sodium tripolyphosphate, 5.5% by weight of a sodium salt of nitrilotriacetic acid, 22% by weight of sodium sulphate, 1.5% by weight of carboxymethyl cellulose, 20% by weight of sodium carbonate, 0.3% by weight of optically
95 whitening agent, 3% by weight of nonylpolyglycol ether, 0.5% by weight of alkaline protease with activity of 300 000 Delph units, which detergent was mixed with 0.3% by weight of perfumed carrier which contained porous hydrated silicon
100 dioxide and perfumed composition in weight proportion 1:1. Perfume composition used was prepared on the basis of geraniol, geranyl acetate, terpineol, diphenyl oxide and other aromatic ingredients. Thus prepared washing detergent
105 retained even after 12 months of storing the original intensity and character of scent of the perfume composition used, both in solid state and after dissolution in water.

110 Example 5

A powdered washing detergent was prepared in a manner similar to that described in Example 4, which detergent had the following
115 composition: 8% by weight of sodium dodecylbenzene sulphonamide, 5% by weight of a hard soap of higher fatty acids, 10% by weight of sodium carbonate, 30% by weight of sodium tripolyphosphate, 18% by weight of sodium sulphate, 19% by weight of sodium perborate,
120 1.5% by weight of carboxymethyl cellulose, 4% by weight of sodium silicate, 2.5% by weight of ethylene oxide and propylene oxide copolymer, 0.3% by weight of optically whitening agent, which detergent was mixed with 0.5% by weight
125 of a perfumed carrier which contained porous

hydrated silicon dioxide and a perfume composition in weight proportion 3:1. The constitution of the perfume composition was the same as in Example 4. Properties of thus prepared washing detergent were the same as in Example 4 as regards quality and stability of the perfume after 12 months of storing.

Example 6

A homogeneous powdered soaking and pre-washing agent was prepared by commonly used drying technology in an atomizing tower using heat, which agent had the following composition: 50% by weight of sodium carbonate, 2% by weight of sodium dodecylbenzene sulphonate, 2% by weight of sodium lauryl sulphonate, 10% by weight of sodium tripolyphosphate, 10% by weight of sodium silicate, 4% by weight of sodium pyrophosphate. The obtained detergent was homogenized with 0.3% by weight of a perfumed sorbent which contained porous hydrated silicon dioxide and a perfume composition on the basis of eucalyptus oil in weight proportion 5:1. Thus prepared soaking and washing detergent retained the intensity and character of the perfume used after 12 months storing, both in solid state and in an aqueous solution.

Example 7

A powdered cleaning agent was prepared by mixing technology in a homogenization drum, which agent had the following composition: 4% by weight of sodium dodecylbenzene sulphonate, 2% by weight of sodium tripolyphosphate, 17% by weight of sodium pyrophosphate, 32% by weight of sodium sulphate, 35% by weight of sodium carbonate, 4.5% by weight of sodium silicate and 0.3% by weight of a perfumed carrier which contained porous hydrated silicon dioxide and a perfume composition in weight proportion 4:3. The used odiferous composition was on the basis of limonene, dipentene and veratrene. As regards both quality and intensity of perfumation the properties of the thus prepared powdered cleaning detergent were after 12 months of storing on the same level as immediately after preparation.

Example 8

97% by weight of a powdered detergent prepared in a manner similar to that described as in Example 1 was mixed with 0.3% by weight of a perfumed sorbent which contained 67% by weight of a porous sorbent on the basis of alkaline polyamide—6 and 33% by weight of the perfume composition on the basis of limonene, dipentene and veratrene. A homogeneous washing agent with intensive and characteristic scent was obtained. As regards perfumation intensity the quality of the thus prepared washing agent was after 8 months of storing the same as after preparation.

Example 9

97% by weight of a powdered detergent

prepared in the same manner as in Example 1 was mixed with 0.3% by weight of a perfumed sorbent which contained 67% by weight of a porous sorbent on the basis of polyphenylene oxide and 33% by weight of the perfume composition on the basis of limonene, dipentene and veratrene. A homogeneous washing agent with an intensive and characteristic scent was obtained, which scent retained the same intensity after 10 months of storing.

Example 10

97% by weight of a powdered detergent prepared in a manner analogous to that described in Example 1 was mixed with 0.3% by weight of a perfumed sorbent which contained 67% by weight of a porous sorbent on the basis of acrylonitrile, butadiene and styrene copolymers (ABS polymer) and 33% by weight of a perfume composition on the basis of limonene, dipentene and veratrene. A homogeneous washing agent with an intensive and characteristic scent was obtained. The intensity of said scent did not change even after 12 months of storing.

Example 11

50% by weight of porous hydrated silicon dioxide having a specific surface of $110 \text{ m}^2/\text{g}$ was mixed with 50% by weight of a perfume composition on the basis of eucalyptus oil, benzoate and toluate derivatives, terpinylacetates, dehydrolinacetate and aldehydes and ketones and their derivatives, the maximum sorption of the composition on a given sorbent being 2.2 weight parts of perfume to 1 weight part of sorbent. A powdered immobilized perfume with the characteristic scent of the original perfume was obtained after homogenization; the intensity of the perfume remained unchanged after 12 months of storing.

Example 12

67% by weight of a porous sorbent on the basis of polyethylene terephthalate having a specific surface of $70 \text{ m}^2/\text{g}$ was mixed with 33% by weight of a perfume composition on the basis of limonene, dipentene and veratrene, the maximum sorption of the composition on a given sorbent being 1.6 weight parts of perfume to 1 weight part of sorbent. A powdered immobilized perfume of the same properties and stability after long storing as in Example 1 was obtained after homogenization.

The process for producing detergents according to the invention is especially suitable for producing powdered detergents with a stabilized perfume ingredient. The process will find use in the manufacture of pre-washing, soaking and washing agents, and perfumed cosmetic and deodorizing preparations in powdered form.

Claims

1. A method of manufacture of perfumed detergents, in which the ingredients of the

detergent are mixed in the desired ratio, a perfume is added thereto and the mixture is homogenized, wherein before its addition to the other ingredients of the mixture, the perfume is

- 5 sorbed on a porous carrier and the thus
Immobilized perfume is added in the amount of 0.05 to 10% by weight, as related to detergent.

2. A method according to Claim 1, wherein the immobilized perfume is added in the amount of

- 10 0.2 to 1% by weight.

3. A method according to Claim 1, wherein the porous carrier is a sorbent with a large active surface, said sorbent being on the basis of polyacrylates, polyvinyl chloride, polyalkyl

- 15 methacrylate, acetylcellulose, polyphenylene

oxides, polyethylene terephthalate, polyamides, urea-formaldehyde and melamine-formaldehyde resins, copolymers of acrylonitrile, butadiene and styrene, polyacrylonitrile, copolymer of vinyl chloride and vinyl acetate, polyethylene, polypropylene, polystyrene, hydrated silicon dioxide, titanium dioxide, or aluminium trioxide, or their mixture, the perfume content in this sorbent being 10 to 65% by weight.

- 20
25 4. A method of manufacture of perfumed detergents, substantially as herein described with reference to the Examples.

5. A detergent made by, or with the use of, a method according to any one of Claims 1 to 4.

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